

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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In re the Application of: Yasushi NAKAYAMA et al.

Serial No.10/526,906

Group Art Unit: 1713

Filed: October 13, 2005

Examiner: Caixia Lu

For: MAGNESIUM-CONTAINING CARRIER COMPONENT AND APPLICATION TO
OLEFIN POLYMERIZATION

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The Honorable Commissioner of Patents and Trademarks
United States Patent and Trademark Office
Alexandria, VA 22313-1450

DECLARATION UNDER 37 CFR 1.132

Sir:

I, Yasushi Nakayama, declare and state that:

1. In March 1993, I was graduated from the master course of Tokyo Institute of Technology, department of Environment Chemistry and Engineering, majoring in Industrial Chemistry, and received a degree of master of Engineering from the same University. Since 1993, I have been an employee of Mitsui Petrochemical Industries, Ltd. (now Mitsui Chemicals, Inc.) and till the present time I have been engaged in Research of olefin polymerization catalyst.

2. I am a co-inventor of the invention described in the specification of the above-identified application.

3. The following experiment was carried out in order to demonstrate

the superiority of the presently claimed invention.

[Additional Comparative Example 1]

Component (c1-8) was prepared in the same manner as Comparative Example 1.

Preparation of component (b1)

95.2g (1.0 mole) anhydrous magnesium chloride, 442 ml decane and 390.6 g (3.0 moles) 2-ethylhexyl alcohol were reacted at 130°C for 2 hours to give a uniform solution (component (b1)).

Preparation of component (b1-8)

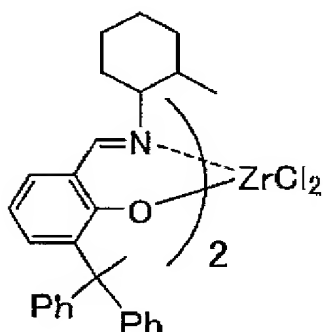
A flask having an internal volume of 200 ml purged sufficiently with nitrogen was charged with 25ml component (b1) (25 mmol in terms of magnesium atom) and 100 ml purified decane, and while the temperature of the solution was kept at 15°C under stirring, 26mmol triethyl aluminum diluted with purified decane was added dropwise thereto over 30 minutes. Thereafter, the temperature of the solution was increased to 80°C over 2 hours, and the mixture was reacted for additional 1 hour. After the reaction was finished, the solids were collected by filtration and washed sufficiently with toluene, and 100 ml toluene was added thereto to form a toluene slurry of component (b1-8).

A part of the component (b1-8) prepared by the above procedure was dried and examined for its composition, indicating 6.5 weight% magnesium, 3.5 weight% aluminum, 78.0 weight% 2-ethylhexoxy group and 18.0 weight% chlorine. Accordingly, the molar ratio of magnesium to aluminum (Mg/Al) was 2.0, and the molar ratio of 2-ethylhexoxy group to aluminum (OR/Al) was 4.6.

Preparation of component (b1-8-a2-172)

A flask having an internal volume of 200 ml purged sufficiently with nitrogen was charged with component (b1-8) in an amount of 10

A part of the of component (b1-8-a2-172) prepared by the above procedure was dried and examined its concentration, indicating 6.5 weight% magnesium, 3.5 weight% aluminum, 77.0 weight% 2-ethylhexoxy group, 18.0 weight% chlorine, and 0.006 weight% zirconium. Accordingly, the proportion of supported zirconium (= the amount of zirconium which contained in the solid after preparation / the amount of zirconium which was used in the preparation $\times 100 \%$) was as very low as 1.3 %, the molar ratio of magnesium to aluminum (Mg/Al) was 2.0, the molar ratio of 2-ethylhexoxy group to aluminum (OR/Al) was 4.6, and the molar ratio of magnesium to zirconium (Mg/Zr) was 4070.



Polymerization of ethylene was carried out in the same manner as Example 1 except that in the polymerization, the component (b1-8-a2-172) prepared above was used in place of the component (b1-1-a2-172).

500 ml purified heptane was introduced into an SUS autoclave having an internal volume of 1 liter purged sufficiently with nitrogen.

and the liquid phase and gaseous phase were saturated with ethylene by blowing ethylene. Thereafter, the heptane was heated to 75°C and the autoclave was charged in an ethylene atmosphere with 0.5 mmol triethyl aluminum and the above component (b1-8-a2-172) (0.0004 mmol in terms of zirconium atom) in this order. Polymerization was carried out for 60 minutes at an ethylene pressure of 0.78 MPa_g. During the polymerization, the system was kept at 80°C and at an ethylene pressure of 0.78 MPa_g. After the polymerization was finished, the reaction product was washed with a large amount of hexane and collected by filtration with a glass filter. The resulting polymer was dried under vacuum for 10 hours to give 0.07 g polyethylene.

The polymerization activity was as very low as 0.18 kg/mmol-Zr₂hr, and the polymerization activity per g of the component (b1-8-a2-172) was also as low as 0.0001 kg/g-cat₂hr.

4. From the results of the above experiment, and based on the my knowledge and experience on Polymer Chemistry, I conclude that:

The molar ratio of alkoxy group to aluminum (OR/Al) defined in the present claim was essential to attain the objective of the present invention.

The undersigned declares further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Respectfully submitted,

this 9th day of January, 2007

Yasushi Nakayama
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